11.5T Superconducting Magnet and Dilution Fridge

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11.5T Dilution Refrigerator User Manual

This system is complicated, easy to damage and expensive to fix. For these reasons it should be operated only under the watchful eye of an experienced user. This manual is designed to be an aid to an occasional user who is comfortable with the general workings of the system, but needs a quick reminder of some of the details. Please do not attempt to learn something new without some supervision and contact Evan Fitzgerald (x6657, evan@nist.gov) with any questions about the system or this manual.

I. 4.2K Cool Down

This process is normally done by Evan Fitzgerald and not the users of the system. Coordination with him as well as proper notification through the instrument schedule webpage is an essential part of any experiment. This process will be described here for information purposes only.

The 4.2K cool down process is the most complicated, as well as the most critical, procedure in the running of this system. Two full days should be allotted for this step. The sample loading procedure can be done before, or basically at any point during the cool down and is described in detail in section II.

1. It is a good idea to do a room temperature sensor check. This means inserting the slug into the sample position and verifying sensible resistance readings for all sensors. See section II for slug loading procedure.
2. After connecting the hose to the gate valve on the top of the cryostat use the turbo pump to evacuate the line then open the gate valve and baffles to pump out the IVC. Once the IVC is on the $1x10^{-5}$ mBar scale open the valve to the OVC and let those both pump down as long as possible. When you stop pumping close the vacuum valves and the baffles.
3. Pump out the 1K pot using the ^4He pump, stop the pump and backfill the pot with He gas connected to the hose barb on the back of the IGH then through valves 2A, 5A and 4A. Repeat this process three times, the last time leaving the pot pumped out.
4. The lambda frig must also be pumped out. Make sure the large valve above the lambda frig pump and a small valve leading into the pumping line are closed, then turn on the pump and slowly open the large valve. This space will pump out quickly (< 5 min.) and the pressure can be monitored on the Lambda Controller front panel by pressing the "display sensor" button until the LED next to "Pressure" is lit. The large valve should be closed before turning the pump off.
5. With the LHe dewar exhaust unrestricted start filling the LHe dewar with LN_2.
6. While that is filling add some He exchange gas to the IVC. About two feet of rubber tubing at ~5psi can be added through the small IVC port on the top of the cryostat. If there is *not* any exchange gas present the thermal mass inside the IVC

will not cool. This can be monitored with the sorb sensor that has a maximum reading of 249.9K.

7. After about half an hour start filling the LN_2 dewar with a second storage dewar until it is full. When it is full stop filling the LHe dewar as well and use a bakelite rod to measure the LN_2 level. There should be at least 4-5 inches in the LHe dewar to last overnight.
8. As long as the sorb sensor is cooling the system can be left overnight to cool.
9. The next morning the entire system should be near 77K and the LN_2 can be blown out of the LHe dewar. Pressurize the dewar through the He exhaust port and use the small section of the fill line inserted into the fill port as the exit path. The LN_2 should be caught in a small dewar and then disposed of outside, so it is not just splashing on the floor. Depending on the amount left in the dewar overnight this can take 0.5 – 1.5 hrs.
10. Once the LN_2 stops flowing seal the He dewar and pump it down using the ^4He pump through valves 4A and 1A. If LN_2 is present the pressure will hang up at 15mbar and step 9 needs to be continued. If the pressure steadily drops below 15mbar let the pressure bottom out, stop the pump and fill the dewar with He gas to ~900mbar. This can be done by connecting He gas to the hose barb on the back of the IGH labeled “vent” and then opening valves 2A, 5A4A and 1A. Pump the dewar down again and repeat the back-fill with He gas. The dewar should be pumped out a total of three times and left full of He gas.
11. With the He dewar full of gas test the 1K pot and lambda frig. needle valves to make sure they open and close. If either is stuck shut remove the blue plastic

cover on the top of the cryostat, loosen the small set screw in the aluminum base and turn the entire assembly counter clockwise a quarter turn. Continue checking the valve after every quarter turn until it operates normally.

12. Start filling the LHe dewar by inserting the fill line completely and using a storage dewar pressure of < 3 psi. This puts the LHe directly around the magnet and cools it most efficiently. After the magnet has been 4.2 K for a few minutes the fill line can be pulled up 3-4 inches and the storage dewar pressure increased to ~ 6 psi to finish the fill. Once the LHe starts collecting the LN_2 should also be topped off if necessary. If you are not filling LN_2 be sure to cover both the exhaust ports when filling the LHe so air does not get sucked in. The entire He fill should take 1.5 – 2.5 hrs.
13. The system once again simply needs to cool overnight, but this time the sorb temperature should be set to 40K so the He exchange gas does not get pumped into it.

II. Sample Loading/Unloading

As seen in the sample mounting information there are two ways to load a sample: top and bottom. Bottom loading should only be used as a last resort when the sample cannot fit the 1” top loading diameter restriction. Only top loading will be described here because it is the normal method. A sample can be loaded or unloaded anytime the mixture is securely in the dump, but it is easiest at room temperature. The sample loading stick is very long and should be stored on its holder inside the blue cage in the High Bay.

1. If unloading skip to step 2. If loading attach the slug to the *left-handed* threads on the stick finger-tight then tighten with the torque wrench to 15 Nm. The lower half of the slug should just be finger-tight, no more, and then the sample can be threaded on the bottom with some grease for better thermal contact. Mark the location of the large key on the side of the slug using the tape on the top of the stick. Pull the metal shroud down as far as it will go over the slug then lock the stick in place with the clamp. If the sample is too long to fit inside the shroud loosen the two screws on the hard plastic stop above the slug and slide it towards the slug until it will fit.
2. To provide exchange gas to cool the stick on its way in set the sorb temperature to 40K if the system is cold. If the system is warm He gas is either present in, or needs to be added to, the IVC. To add He gas start flow through a rubber tube, connect it to the hose barb on the IVC connection after purging, pinch off the tube about a foot away from the barb then open the valve briefly so the gas is sucked in.
3. Remove the blank off flange above the gate valve on the top of the cryostat and mount the stick. This can be done by one person, but requires some practice, so two sets of hands will be useful the first few times. Attach the IVC pumping line to the lower port on the side of the metal shroud and pump it out using the turbo down to the 10^{-5} scale.
4. Once the shroud has been pumped out valve off the turbo pump so you don't pump away the exchange gas and then open the gate valve. It is normal to hear a quick sucking noise, but a lengthy one or an increased helium boil-off are signs

that something is wrong. Open the baffles by turning the knob counter clockwise as far as possible, about a quarter turn. The stick can now be lowered into the IVC by removing the clamp holding it above the shroud. If it does not slide easily rub the length of the stick with some vacuum grease.

5. If the IVC is cold the stick should be brought close to the same temperature before reaching the sample position. After sliding the stick in about half way clamp it off and let it sit for about 30 minutes then lower it in about ¾ of the way and leave it for another 30 minutes.
6. If you are loading skip to step 7. If you are unloading push the stick in until it touches the top of the slug, ~ 46.5mm gap from the top of the shroud to the first change in diameter up the stick, and leave it in this position for about 15 minutes. Turn the stick counter clockwise until there is a sudden increase in resistance, ~ 6 turns and gap of 35mm, then keep turning 9 more full rotations until the whole assembly can be pulled out. Pull the stick up, warm it when the o-ring freezes, continue pulling and warming then clamp it into position and close off the gate valve when all the way up. Skip to step 8.
7. Line the piece of tape on the top of the stick up with the sticker marking the key slot on the top of the gate valve and lower the stick into the slot until the gap from the top of the shroud to the first increase in diameter up the stick is 67mm. Then turn the stick clockwise about three turns until it drops slightly and the gap is 44mm. Continue turning about 8.5 turns until the slug breaks loose. It is tight and kind of violent! Keep going for about another 6 turns at which point the stick can

be pulled all the way up after warming the frozen o-ring a few times then clamped and the gate valve closed.

8. At this point the stick, with or without a sample, can be warmed quickly by breaking the vacuum using the valve above the turbo pump. If, however, the sample is sensitive to abrupt temperature changes the sample can be left on the stick in vacuum to slowly warm up. It is also possible to add helium gas to the shroud to speed the warm up time with out condensing anything on the slug and sample. Be aware of the height of the stick, especially when in C-100, and leave proper warning for others in the area if it is being left unattended.

III. Base Temperature Cool Down

The majority of this process is done under the control of multiple LabVIEW programs written by Oxford Instruments for this specific piece of equipment. These programs in general do a good job and should be utilized to make your life easier. Even though the programs will be doing most of the work I will try to explain what should be happening at each step.

1. To attempt a cool down to base temperature the IVC should be at or near 4.2K, the sample temperature should not be more than 15K, the magnet can be energized or not and the LHe and LN_2, should be full.
2. Fill the cold trap on the back of the equipment cart with LN_2, close the baffles using the knob on the top of the cryostat near the He fill port and close the gate valve on top of the IVC.
3. From the main menu press the “Kelvinox” button and when the VI opens, a dialog box appears asking if you would like to turn the pumps on. Feel free to select

"OK" unless you are not going to start the cool down immediately. Next select the "gas flow" button in the Kelvinox VI. This will open a VI that looks like the IGH front panel and allows you to control all of the valves, heaters and pumps without disrupting communication.

4. Press the "Fill 1K Pot" button and select "OK" when the dialog box, or boxes if the pumps are not on, appears. As it says this will fill the 1K pot to cool the sorb then determine and set the correct needle valve setting to keep the pot cold. Although this is all done automatically you must verify that the needle valve is opening. Watch the pressure on G3 when the VI says it is filling the pot and make sure it rises at a quick, steady pace. If the valve is stuck refer to the procedure in step 11 of the 4.2K Cool Down in this manual. This process should take around half an hour and when it is finished the VI will simply close. What will continue running is a VI that monitors the 1K pot and keeps it below 2K by opening or closing the needle valve. This VI can be stopped or started by pressing the button under "Monitor" in the 1K pot section of the Kelvinox VI. A green light is lit next to the button when it is monitoring.
5. Next press the "Condense In" button and when the dialog box appears open the manual dump valves, open the valve labeled "condenser" on top of the cryostat and then press "OK". This VI will slowly open valve 12A to allow the mixture into the dilution unit where it is condensed by the 1K pot. When this VI is done it will again simply close.
6. Finally press the "Circulate" button and then "OK" when the dialog box pops up. This VI will slowly open valve 6 to begin circulation of the mixture through the

dilution unit. When this VI is finished it will close just like the others. The frig. will be left running and should be cooling towards base temperature. P1 will soon be less than 1mbar and G1 and G2 should be approximately equal. If there is a large pressure differential across the cold trap it may be blocked and you can switch to the other trap by pressing the “Change LN_2 Cold Trap” button.

7. The only setting under user control that can affect the base temperature is the still heater. By changing the amount of heat added to the still you control the rate that the ^{3}He is pumped away. The “circulate” VI will turn on the still heater to around 3mW which should work fine. A slightly lower base temperature may be achieved with either a higher or lower setting, but the change should not be more than one or two milli-watts.

IV. Temperature Control

This system is designed for use at temperatures below 1K, however it is possible to reach temperatures of around 10K. Controlling across this wide temperature range is a matter of finding the correct settings on the temperature controller. Figuring out these settings can be challenging, but hopefully with these guidelines things will be a bit easier.

1. Use an appropriate sensor: MC Slug is closest to the sample position and should be used for control below 2K; Cernox is the only well calibrated sensor above 2K.
2. Use the appropriate amount of cooling power: < 1K operate the fridge normally as if you were going to base temperature; 1.0 – 1.8 K leave valve 9 open with the mixture still circulating as a buffer to take extra mixture and decrease cooling power, > 1.8 K turn off the ^{3}He pump to stop circulation and further decrease

cooling power, but leave the mixture in the unit and run the 1K pot as normal for some cooling power.

3. Setting up the temperature controller: The gain should be set to the lowest setting, T_I should be set to P_D, T_D and Bias should be zero and Range should be on the lowest setting. The error switch on the left, as well as the heater output switch on the far right, should be set to short.
4. Manually setting a target resistance: Setup the AVS monitor VI to display only the control sensor using appropriate settings for Range and Excitation. The set point on the temperature controller, the push-button analog display, is read exactly as the digital screen on the AVS and can be set to the desired resistance. Then hit the small enter button to register it.
5. Start controlling: The error switch can now be moved to the normal position and a nonzero value on the error sweep gauge should be visible. If the error reading seems reasonable then turn the mixing chamber heater on by pressing the on/off button on the IGH controller and then selecting a power range. The heater output on the AVS should then be switched to normal and an appropriate range on the AVS selected by watching the output sweep gauge. The change in the temperature can be seen on the AVS monitor program. Note: The AVS monitor VI's implementation of temperature control is very temperamental and most users who attempt to use it end up going back to manual control.
6. Fine-tuning control: The gain value should be increased until the heater output is enough to reach the set point. However, if the heater output is maxed out, all the way to the right on the sweep gauge, then the range should be increased before the

gain. Do not forget that the range is limited by whichever range setting is lower the AVS or the IGH (the maximum output of 20mW by the IGH can be circumvented when more power is needed, see step 7). Once the set point has been reached there may be no further settings necessary depending on the accuracy needed. If after an initial overshoot there is an unacceptable oscillation around the set point then set T_I equal to the period of oscillation in seconds.

7. High temperature control: As mentioned in step 6 the normal maximum heater output is 20mW this is because the system is designed for low temperature control. To work at higher temperatures the IGH is circumvented by turning the IGH mixing chamber heater off, switching the AVS heater output to "short", unplugging the red and black plugs from the back of the temperature controller, plugging in the black and brown connections and unplugging the connector labeled "heaters" on the back of the IGH. The temperature controller heater output now goes directly into the cryostat instead of going through the IGH. In this case only the range setting on the AVS limits the output.

V. Computer Control Using ICP

Computer control is now available at all instruments running ICP. This interface doesn't talk directly to the power supply or the temperature controller as with other systems. Instead ICP communicates with a LabVIEW program running on the system computer, which then interfaces with the different parts of the system. This difference is transparent to the user when in operation, however, the startup is not.

1. The iomain.vi should already be running with the pot and quench monitors both on if necessary. It is probably a good idea to shutdown any vi's that are not in

use. One that cannot be running if you are going to remotely check the temperature is the AVS Monitor. Stop it by either hitting the disconnect button and waiting for the sensors to cycle through and stop or by simply selecting close under the file menu.

2. Double-click on the ICP-new.vi icon on the desktop to open the program that will directly communicate with ICP. The front panel will appear, but the program will not run. Change the sensor, range, and excitation to the correct values for the sensor you wish to monitor and/or use for control and change the update settings switch to yes. Also make sure that the AVS, directly above the screen, is set to auto-range or else you will not be able to remotely change the range. Even though the setting is called auto range all changes in the range must be input by the user and will not happen automatically.
3. Press the run icon, a single arrow pointing to the right, to start the program. The AVS should be set to the sensor and settings you entered. The program is now waiting for a command from ICP and any changes to settings on the front panel will not take affect until a command is received.
4. The system computer communicates to all of the components through the magnet controller. If communication is lost, GPIB error asking you to abort, the programs running must be shutdown, the magnet controller’s power cycled then the programs restarted. All of the system components should be in remote mode to allow them to communicate.
5. Now that the system computer is ready lets move back towards the instrument computer. First plug the temperature controller RS232 cable into the back of the

system computer. Then type “icp” at the command prompt on the instrument computer. Once ICP has started type “tdev” and follow the commands for setting up the Oxford 12T temperature controller. Next type “hdev” and follow the commands for setting up the Oxford 12T magnet controller. After these have both been setup type in “pt” to print the temperature and “phf” to print the field and verify that they are correct.

6. The magnet can be controlled by using the command “sm= x” where x is the desired field. The program will determine the current status of the magnet and safely change the field to the new value. Once the field is reached and the magnet is persistent, if persistent was set to true, control is returned to ICP.

VI. Warm-Up From Base Temperature

The warm-up or make safe procedure is generally done at the end of an experiment to put the system in a safe mode to move off of the instrument. It is relatively simple and, as with other things, done almost completely by a LabVIEW VI. This process generally takes approximately one hour before the system is safe to move. Be sure to schedule your time accordingly because not completing this process leaves the mixture vulnerable to escape and is an unacceptable state to move the system in.

1. Press the Make Safe button on the Kelvinox front panel to begin the process. This should open valve 3, close valves 13a and 13b and open valve 9 while leaving the pump running. You will need to open the manual dump valves on the top of the dump.
2. Monitor the pressure on G2 and compare this to the dump pressure at the start of the experiment, or end of the last experiment. When these two values match and

P1 is less than 0.1 mbar valves 12a and 12b can be closed followed by valve 3, then valve 6, then the manual dump valves and finally valve 9. The pump can be turned off and the system is now safe.